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Fumigation of Natural Raisins With Phosphine

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This publication reports research involving pesticides. It does not contain recommendations for their use. All uses of pesticides must be registered by appropriate State and/or Federal agencies before they can be recommended.

CAUTION: Pesticides can be injurious to humans, domestic animals, desirable plants, and fish or other wildlife—if they are not handled or applied properly. Use all pesticides selectively and carefully. Follow recommended practices for the disposal of surplus pesticides and pesticide containers.



ACKNOWLEDGMENTS

A number of individuals and groups cooperated with the Agricultural Research Service in this study. Floyd Leon Bell, laboratory helper, Agricultural Research Service, Stored-Product Insects Research Branch, Dried Fruit and Tree Nut Insects Investigations, at Fresno, Calif., assisted in the studies by preparing the test insects, assisting in placing test insects and fumigant sampling tubes in stacks, taking fumigant concentration readings, and making insect mortality counts.

The Sun-Maid Raisin Growers of California provided the raisins, sisalkraft paper, labor, and other material used for forming and covering the stacks.

The Hollywood Termite Control Company, Inc., Alhambra, Calif., supplied the aluminum phosphide tablets from which the phosphine evolved and equipment for measuring the fumigant concentrations in the stacks.

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Fumigation of Natural Raisins With Phosphine

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BACKGROUND OF THE STUDY

From 240,000 to 250,000 tons of natural raisins are produced annually and stored in the central California area. Approximately 75 percent of these are stored out of doors on blacktop pavement in stacks covered with sisalkraft paper. The raisins remain in storage until they are processed; some are stored for 1 year or more.

Raisins may be infested with insects at the time they are removed from the drying trays in the vineyards or they may become infested during storage. To eliminate the insect infestation, the raisins are fumigated at the time they are placed in storage and at intervals while in storage.

During the past few years, phosphine has been used as a fumigant in the United States. It is manufactured in tablet and pellet form and is composed mainly of aluminum phosphide and

ammonium carbamate. When exposed to the atmosphere, the tablets or pellets decompose to form phosphine (hydrogen phosphide), ammonia, carbon dioxide, and aluminum hydroxide. Phosphine is a gas that is slightly heavier than air, has an odor resembling carbide, and is effective against insects.

Four tests were made to determine the effectiveness of phosphine against the most common insects that attack natural raisins stored in stacks covered with sisalkraft paper.

The Food and Drug Administration¹ has established a residue tolerance of 0.01 parts per million (p.p.m.) for the use of this fumigant on processed foods, one of which is raisins. Previous results with the type of treatments discussed in this study indicate that residues greater than 0.01 p.p.m. would not be expected.

TEST PROCEDURE

General

In each test, natural raisins were placed in sweatboxes and stacked, and 1- by 3-inch stringers were nailed to the outsides of the stacked sweatboxes. Two layers of sisalkraft paper were nailed to the stringers and held in place with wooden lath. Each stack was covered so as to form a rounded dome, which facilitated water runoff and created an airspace above the raisins (fig. 1). The surface of the outer layer of paper was treated with a thin layer of white plastic to shed water and reflect the heat. The edge of the paper around the bottom of the stacks was held against the

blacktop by a sand-oil mixture (fig. 2). This mixture created a tight seal and also prevented water, insects, and rodents from entering the stacks.

Air samples were drawn through ¼-inch polyethylene tubing, and the concentrations were determined by drager tubes (fig. 3).

The fumigant tablets were placed on paper plates, which were distributed along the top center of the stacks of raisins.

Insects were placed in separate test cages at each location. These cages were made of

¹ Federal Register, v. 32, No. 176. Tuesday, Sept. 12, 1967.

small plastic vials with brass screen inserted in the lids to allow the gas-air mixture to reach the insects. A small amount of food was placed in each cage with the insects.

The exposure period for small stacks in all tests was 6 days. At the end of this period, the test insects were removed.

For each test, temperature and relative humidity records were obtained by a hygro-thermograph placed in one stack during the exposure period. In addition, for all but the first test, a dial thermometer was inserted near the edge of one stack, and readings were taken when the fumigant samples were obtained.

Test 1

Four stacks of natural raisins were used in test 1. Stack 1 occupied 15,000 cubic feet of space and stacks 2, 3, and 4, 700 cubic feet each.

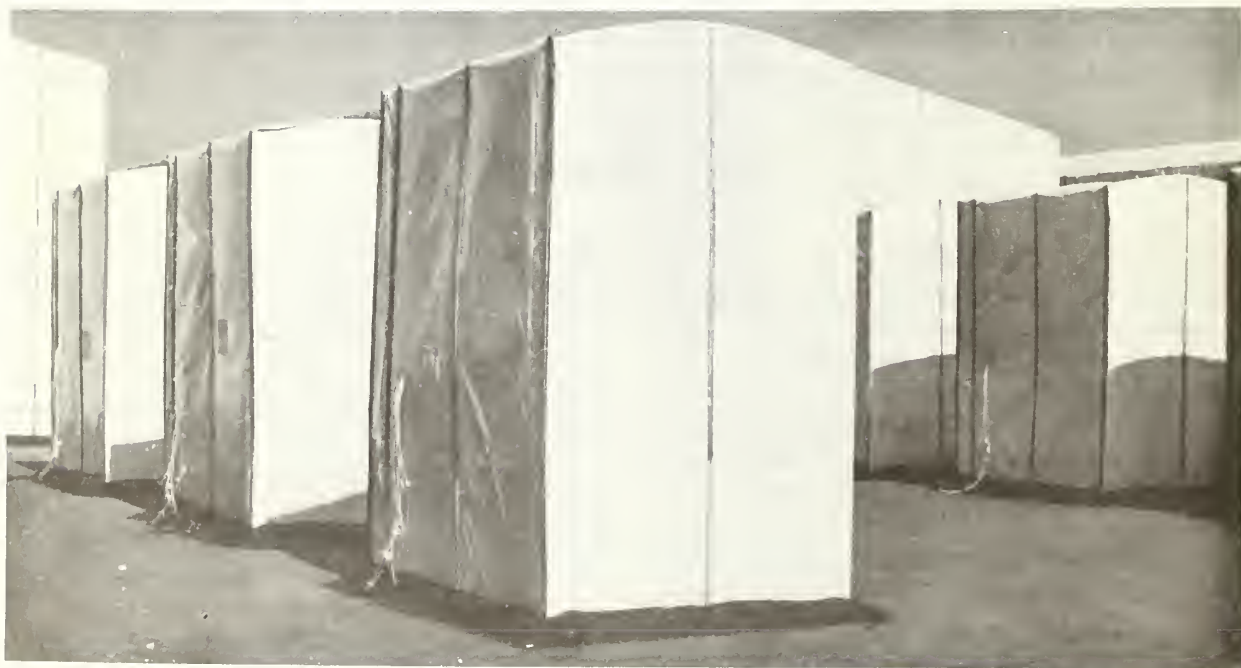
Stack 1 was fumigated with phosphine at the rate of 45 tablets of aluminum phosphide per 1,000 cubic feet. The three smaller stacks were fumigated at the same time as the larger

stack at the rates of 40, 35, and 30 tablets per 1,000 cubic feet, respectively.



BN-36427

FIGURE 1.—Stacks of raisins being covered with sisal-kraft paper.



BN-36428

FIGURE 2.—Small and large stacks of natural raisins covered with sisal-kraft paper and sealed to blacktop with a mixture of sand and oil.



BN-36429

FIGURE 3.—View of polyethylene tubing through which fumigant samples were taken.

At weekly intervals, a perimeter treatment was applied to the outside of stack 1 and to the blacktop area within 10 feet of the stack. A 4-percent diazinon emulsion concentrate, diluted with water according to manufacturer's directions, was sprayed on the entire outer surface of the stack to the point of runoff. The blacktop surrounding the stack was thoroughly wet with the spray.

Test insects were placed at 48 locations in stack 1. These included adults of the merchant grain beetle, *Oryzaephilus mercator* (Fauvel), and larvae of the Indian-meal moth, *Plodia interpunctella* (Hübner). At each location, 25 merchant grain beetles and 10 Indian-meal moth larvae were placed in separate cages.

Test insects were placed at six locations within each small stack. One vial containing 25 adult merchant grain beetles and one containing 10 larvae of the Indian-meal moth were placed at each location.

In test 1, 20 samples containing adult merchant grain beetles and 20 containing Indian-meal moth larvae were used as controls for all four stacks and held under the same environment as those in the fumigated stacks.

Fumigant samples were drawn from 36 locations within the enclosed large stack. The samples were taken at 8:00 a.m. and 4:00 p.m. for the first 3 days and at 4:00 p.m. during the next 3 days. Samples were taken at 3- to 9-day intervals during the remaining 46 days. The fumigation began on November 15, 1967, at 5:00 p.m. and ended January 4, 1968, at 4:00 p.m.

Fumigant samples were drawn from six locations within each of the small stacks. Two readings were made from each location from each stack during the first 3 days. These were taken at 8:00 a.m. and 4:00 p.m. One sample was taken at 4:00 p.m. on each of the remaining 3 days.

At monthly intervals, fourteen 1-quart samples of raisins were removed from predetermined locations within the large stack. Each sample was then examined for the presence, abundance, and species of living insects.

Test 2

In test 2, five small stacks of natural raisins were used. Stacks occupied 700 cubic feet each and were fumigated with phosphine at the rates of 30, 25, 20, 15, and 10 tablets per 1,000 cubic feet. The fumigation began on February 6, 1968. In this test, placement of insect samples and species and stages of insects used were the same as for the small stacks described in test 1. At each location, 100 merchant grain beetle adults and 15 Indian-meal moth larvae were placed in separate cages. The same numbers and kinds of insects were placed in a stack that was not fumigated and were used as controls.

Test 3

Seven small stacks of natural raisins were used in test 3, and each contained 700 cubic feet of space. Six of the stacks were fumigated with phosphine, and one stack remained untreated. The dosages used were 30, 25, 20, 15, 10, and five tablets per 1,000 cubic feet. Fumi-

gant samples were taken from 10 locations within each stack. Six were located in space, and one was located in the center of the mass of raisins in each of four sweatboxes. The stacks were fumigated on March 21, 1968.

Test insects were placed in each stack at the same 10 positions from which the fumigant samples were taken. Insects were also placed in 10 locations in the untreated stack. At each location, 50 adult merchant grain beetles, 15 Indian-meal moth larvae, and 15 raisin moth larvae, *Cadra figulilella* (Gregson), were placed in separate cages.

Test 4

In test 4, 11 small (700-cubic-foot) stacks of natural raisins were used. Five of the stacks were fumigated with phosphine applied at the rate of five tablets per 1,000 cubic feet and five at the rate of 10 tablets per 1,000 cubic feet. One control stack was not fumigated. Fumigant samples were drawn at the same intervals from three locations in each stack. Stacks were fumigated on April 25, 1968.

The test insects used were the same species and stages as in test 3. Each sample included 50 beetles and 15 larvae of each species of moth.

RESULTS

Test 1

The average concentrations in parts per million of phosphine found in the four stacks during the exposure periods are presented in table 1. Peak concentrations occurred on the

TABLE 1.—*Test 1: Average concentrations of natural raisins fumigated with aluminum phosphide tablets, 1967-68*

Date and time of fumigant-sample reading ¹	Average phosphine concentration			
	Stack 1— 45 tablets/ 1,000 cu. ft.	Stack 2— 40 tablets/ 1,000 cu. ft.	Stack 3— 35 tablets/ 1,000 cu. ft.	Stack 4— 30 tablets/ 1,000 cu. ft.
	<i>P.p.m.</i> ²	<i>P.p.m.</i> ³	<i>P.p.m.</i> ³	<i>P.p.m.</i> ³
11 16 67				
8:00 a.m.	232.9	212.5	225.8	119.2
4:00 p.m.	458.1	445.8	428.3	323.3
11 17 67				
8:00 a.m.	656.2	701.7	590.8	306.7
4:00 p.m.	761.2	666.7	560.8	425.8
11 18 67				
8:00 a.m.	724.0	617.5	475.0	346.7
4:00 p.m.	673.1	583.3	300.0	183.0
11 19 67				
8:00 a.m.	584.0	431.7	256.6	116.7
11 20 67				
8:00 a.m.	463.4	306.7	124.2	85.0
11 21 67				
8:00 a.m.	479.3	293.3	105.0	56.7
11 24 67				
8:00 a.m.	374.5	—	—	—
11 27 67				
8:00 a.m.	271.8	—	—	—
11 30 67				
8:00 a.m.	155.4	—	—	—

TABLE 1.—*Test 1: Average concentrations of natural raisins fumigated with aluminum phosphide tablets, 1967-68—Continued*

Date and time of fumigant-sample reading ¹	Average phosphine concentration			
	Stack 1— 45 tablets/ 1,000 cu. ft.	Stack 2— 40 tablets/ 1,000 cu. ft.	Stack 3— 35 tablets/ 1,000 cu. ft.	Stack 4— 30 tablets/ 1,000 cu. ft.
	<i>P.p.m.</i> ²	<i>P.p.m.</i> ³	<i>P.p.m.</i> ³	<i>P.p.m.</i> ³
12/4/67				
10:00 a.m.	82.5	—	—	—
12/7/67				
10:00 a.m.	62.5	—	—	—
12/11/67				
1:00 p.m.	47.5	—	—	—
12/18/67				
1:00 p.m.	20.0	—	—	—
12/27/67				
10:00 a.m.	8.3	—	—	—
1/4/68				
2:00 p.m.	7.3	—	—	—

¹ Fumigation was started at 5:00 p.m. on 11/15/67.

² Average from 36 sample points.

³ Average from 6 sample points.

second day after application of the aluminum phosphide tablets. At the end of 50 days, measurable amounts of phosphine were present in the 15,000-cubic-foot stack fumigated at the rate of 45 tablets per 1,000 cubic feet. This stack of raisins became reinfested with insects during the 9th month in storage and was fumigated again at the rate of 45 tablets per 1,000 cubic feet of space. The insects found infesting the raisins were saw-toothed grain beetles, *Oryzaephilus surinamensis* (L.).

After the study was completed, it was learned that on at least three occasions the entrance flaps at both ends of the stack had blown open. Complete kills of all caged test insects were obtained from each dosage. A 1.8 percent mortality of the merchant grain beetles and 1.0 percent mortality of Indian-meal moth larvae were observed in the controls. A record of the temperature and relative humidity obtained during the period of this test is given in table 2.

TABLE 2.—*Test 1: Temperature and humidity records obtained by a hygrothermograph placed in a small stack of natural raisins, 1967-68*

Date	Temperature			Relative humidity		
	Mini- mum	Maxi- mum	Mean	Mini- mum	Maxi- mum	Mean
	° F.	° F.	° F.	Percent	Percent	Percent
11/20-27/67	53.0	62.0	57.5	58	64	61.0
12/4-11/67	45.0	55.0	50.0	61	68	64.5
12/11-18/67	39.2	49.5	44.3	66	77	71.5
12/18-26/67	39.2	45.5	42.3	66	73	69.5
12/26/67-1/2/68	41.9	46.4	44.1	61	66	63.5
1/2-8/68	38.3	44.6	41.4	61	68	64.5
1/8-15/68	39.2	45.5	42.3	60	68	64.0
1/15-22/68	41.0	48.2	44.6	59	63	61.0

Test 2

The general pattern of phosphine concentrations observed from the five stacks of natural raisins in test 2 was similar to that obtained in test 1 (table 3). As in the preceding test, each dosage produced a complete kill of the test insects. No insects used as controls died. The maximum and minimum air temperatures observed on the dial thermometer during the test period were 54° and 51° F. (table 6).

Test 3

Test 3 showed that the phosphine penetrated readily into the mass of natural raisins stored in sweatboxes, which were stacked tightly one upon the other. No consistent differences in the concentrations were found in the airspace and within the mass of raisins. The fumigant concentrations are presented in table 4. A complete kill of the test insects was obtained with each dosage used. Insects that were not fumigated lived. Temperature and humidity records are presented in table 6. The max-

TABLE 3.—*Test 2: Average concentrations of phosphine in mass of natural raisins during fumigation, 1968*

Date and time of fumigant- sample reading ¹	Average phosphine concentration				
	Stack 1— 30 tablets/ 1,000 cu. ft.	Stack 2— 25 tablets/ 1,000 cu. ft.	Stack 3— 20 tablets/ 1,000 cu. ft.	Stack 4— 15 tablets/ 1,000 cu. ft.	Stack 5— 10 tablets/ 1,000 cu. ft.
	P.p.m. ²	P.p.m. ²	P.p.m. ²	P.p.m. ²	P.p.m. ²
2/7/68					
8:00 a.m.	161	91	79	80	44
4:00 p.m.	190	130	84	91	75
2/8/68					
8:00 a.m.	313	205	134	141	80
4:00 p.m.	312.5	214	157.5	172.5	97
2/9/68					
8:00 a.m.	354	236.7	207.5	171.7	100
4:00 p.m.	345	202	180	205	106
2/10/68					
8:00 a.m.	326	213.2	205.0	164	95
2/11/68					
8:00 a.m.	131	78.3	109	96	63
2/12/68					
2:00 p.m.	124	69	90	92.5	64

¹ Fumigation was started at 5:00 p.m. on 2 6 68.

² Average from 6 sample points.

imum and minimum air temperatures observed on the dial thermometer during the test period were 63° and 59° F.

Test 4

In order to evaluate more fully the effectiveness of the fumigant at lower dosages, five stacks of natural raisins were fumigated at the rate of five aluminum phosphide tablets per 1,000 cubic feet and five at 10 tablets per 1,000 cubic feet. The dosages used and resulting concentrations of phosphine are presented in table 5. The average concentrations per dosage are found in table 7. The peak concentrations appeared at the end of the second day. Except for the concentrations obtained during the first day, those resulting from the 10-tablet dosage were more than twice as great as those from the five-tablet dosages. Complete kills of all test insects were obtained with each dosage. No insects died in the controls. Temperature and humidity records are presented in table 6. The maximum and minimum air temperatures observed on the dial thermometer during the period of the test were 78° and 64° F.

TABLE 4.—*Test 3: Average concentrations of phosphine in atmosphere around natural raisins in boxes and in airspace over the raisins during fumigation, 1968*

Date and time of fumigant-sample readings	Average phosphine concentration in atmosphere											
	Stack 1—30 tablets/ 1,000 cu. ft.		Stack 2—25 tablets/ 1,000 cu. ft.		Stack 3—20 tablets/ 1,000 cu. ft.		Stack 4—15 tablets/ 1,000 cu. ft.		Stack 5—10 tablets/ 1,000 cu. ft.		Stack 6—5 tablets/ 1,000 cu. ft.	
	Over raisins	Around raisins	Over raisins	Around raisins	Over raisins	Around raisins	Over raisins	Around raisins	Over raisins	Around raisins	Over raisins	Around raisins
	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>
3/22/68												
8:00 a.m.	110.0	110.0	94.2	86.2	71.7	70.0	78.3	56.2	63.3	50.0	33.3	25.0
4:00 p.m.	200.0	195.0	200.0	198.8	130.8	121.2	148.3	100.0	95.0	83.8	55.8	41.2
3/23/68												
8:00 a.m.	260.0	260.0	243.0	226.2	120.8	111.2	130.8	111.2	80.8	90.0	55.0	52.5
4:00 p.m.	264.2	275.0	297.5	301.2	190.8	176.2	149.2	171.2	90.0	95.0	53.3	68.8
3/24/68												
8:00 a.m.	215.0	208.8	290.8	292.5	118.3	127.5	90.8	95.0	68.3	66.2	41.6	36.2
4:00 p.m.	205.8	201.2	285.0	268.8	132.5	117.5	92.5	90.0	59.2	70.0	24.2	26.2
3/25/68												
4:00 p.m.	142.7	141.2	316.7	253.7	112.5	130.0	80.0	85.0	47.5	41.2	21.7	20.0
3/26/68												
4:00 p.m.	72.5	62.5	195.8	178.8	59.2	66.2	43.3	36.2	34.2	27.5	10.0	10.0
3/27/68												
3:00 p.m.	28.3	22.5	113.3	126.2	32.5	25.0	8.3	13.8	—	—	—	—

TABLE 5.—*Test 4: Average concentrations of phosphine in mass of natural raisins during fumigation, 1968*

Date and time of fumigant-sample readings	Average phosphine concentration									
	5 tablets per 1,000 cu. ft.					10 tablets per 1,000 cu. ft.				
	Stack 1	Stack 3	Stack 5	Stack 7	Stack 9	Stack 2	Stack 4	Stack 6	Stack 8	Stack 10
	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>
4/26/68										
8:00 a.m.	25.0	30.0	20.0	11.7	6.7	56.7	40.0	31.7	15.0	40.0
4:00 p.m.	20.0	48.0	48.3	18.3	25.0	61.7	58.0	60.0	33.3	40.0
4/27/68										
8:00 a.m.	36.7	30.0	43.3	31.7	23.3	85.0	80.0	65.0	48.3	61.7
4:00 p.m.	18.3	21.7	23.3	31.7	40.0	83.3	61.7	63.3	73.3	76.7
4/28/68										
8:00 a.m.	15.0	11.7	16.7	23.3	25.0	50.0	58.3	55.0	56.7	65.0
4:00 p.m.	13.3	15.0	16.7	11.7	18.3	51.7	55.0	35.0	28.3	38.3
4/29/68										
8:00 a.m.	11.7	25.0	20.0	21.7	38.3	63.3	51.7	33.3	65.0	60.0
4/30/68										
8:00 a.m.	6.7	8.3	8.3	11.7	20.0	38.3	25.0	13.3	15.0	28.3
5/1/68										
3:00 p.m.	3.3	2.6	.0	3.3	3.3	3.3	3.3	.0	3.3	3.3

TABLE 6.—*Tests 2, 3, and 4: Temperature and humidity records obtained by a hygrothermograph placed in a stack of natural raisins during each test, 1968*

Test No. and date	Temperature			Relative humidity		
	Mini- mum	Maxi- mum	Mean	Mini- mum	Maxi- mum	Mean
	° F.	° F.	° F.	Percent	Percent	Percent
<i>Test 2</i>						
February:						
6	46.4	48.2	47.3	59	66	62.5
7	46.4	48.2	47.3	58	60	59.0
8	47.3	49.1	48.2	—	—	—
9	48.2	48.2	48.2	59	60	59.5
10	46.4	49.1	47.8	59	62	60.5
11	45.5	49.1	47.3	59	61	60.0
12	47.3	50.0	48.6	55	60	57.5
<i>Test 3</i>						
March:						
21	49.1	55.4	52.2	55	62	58.5
22	51.8	55.4	53.6	55	58	56.5
23	52.7	57.2	55.0	54	57	55.5
24	51.8	56.3	54.0	54	58	56.0
25	52.7	55.4	54.0	54	57	55.5
26	49.1	53.6	51.4	56	61	58.5
27	49.1	54.5	51.8	56	61	58.5
<i>Test 4</i>						
April:						
25	52.7	60.8	56.8	55	63	59.0
26	56.3	61.7	59.0	54	59	56.5
27	57.2	62.6	59.9	54	59	56.5
28	58.1	63.5	60.8	54	58	56.0
29	59.0	62.6	60.8	54	56	55.0
30	58.1	64.4	61.2	52	58	55.0
May:						
1	58.1	64.4	61.2	52	58	55.0

TABLE 7.—*Test 4: Average concentrations of phosphine in mass of natural raisins during fumigation, 1968*

Date and time of fumigant- sample readings	Average phosphine concentrations in 5 stacks during exposure period	
	5 tablets 1,000 cu. ft.	10 tablets 1,000 cu. ft.
	P.p.m.	P.p.m.
4/26/68		
8:00 a.m.	18.7	36.7
4:00 p.m.	32.0	50.7
4/27/68		
8:00 a.m.	33.0	70.0
4:00 p.m.	27.0	71.7
4/28/68		
8:00 a.m.	18.3	57.0
4:00 p.m.	15.0	41.7
4/29/68		
8:00 a.m.	23.3	54.7
4/30/68		
8:00 a.m.	11.0	24.0
5/1/68		
3:00 p.m.	2.5	2.7

Summary Table

A summary of the average concentrations resulting from the nine dosages used in the four tests during the 6-day exposure periods is found in table 8.

TABLE 8.—Average concentrations of phosphine found in stacks of natural raisins included in tests 1, 2, 3, and 4

Date and time of fumigant- sample readings	Average phosphine concentration								
	Number of tablets per 1,000 cu. ft.								
	45	40	35	30	25	20	15	10	5
	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>	<i>P.p.m.</i>
<i>1st</i>									
8:00 a.m.	232.9	212.5	225.8	130.3	92.9	75.4	79.2	44.2	22.8
4:00 p.m.	458.1	445.8	428.3	238.0	165.4	107.5	120.0	65.9	38.8
<i>2d</i>									
8:00 a.m.	656.2	707.1	590.8	293.3	224.2	127.5	135.8	74.6	39.3
4:00 p.m.	761.2	666.7	560.8	334.2	255.8	174.2	160.8	81.4	34.5
<i>3d</i>									
8:00 a.m.	724.0	617.5	475.0	305.3	263.8	162.9	131.2	79.1	25.0
4:00 p.m.	673.1	583.3	300.0	244.7	243.3	156.2	148.8	59.8	17.6
<i>4th</i>									
8:00 a.m.	584.0	431.7	256.6	194.7	265.0	158.8	122.1	62.0	22.8
<i>5th</i>									
8:00 a.m.	463.0	306.7	124.2	97.8	137.0	84.2	69.6	34.8	8.8
<i>6th</i>									
3:00 p.m.	479.3	293.3	105.0	69.7	91.2	61.2	50.4	15.7	4.2